CENTESIMAL COMPOSITION AND PHYSICOCHEMICAL PROPERTIES OF OIL EXTRACTED FROM *MORINGA OLEIFERA* LAM SEEDS GROWN IN THE STATE OF SERGIPE, BRAZIL.

Milson Santos Barbosa[a], Cintia Cristina da Costa Freire[a], Danyelle Andrade Mota[a], Lays Carvalho de Almeida[a], Flávia Michelle Silva Wiltshire[a], Lisiane dos Santos Freitas[b], Ranyere Lucena de Souza[a], Matheus Mendonça Pereira[c], Álvaro Silva Lima[a], Cleide Mara Faria Soares[a]

[a] Institute of Technology and Research, Av. Murilo Dantas 300, Aracaju 49032-490, Brazil
[b] Department of Chemistry, Federal University of Sergipe, São Cristóvão 49100-000, Brazil
[c] Aveiro Institute of Materials, Chemistry Department, University of Aveiro, 3810-193 Aveiro, Portugal

Abstract: In this study, the centesimal composition and physicochemical properties of oil extracted from *Moringa oleifera* Lam seeds was investigated. According to results obtained, seeds centesimal composition are constituted by 41% of crude oil, 32.4% of proteins, 4.2% of ash, 19.54% of carbohydrate and 2.86% of humidity. The results indicate that *Moringa oleifera* Lam seeds are a potential source for health and diet food products. Besides, nine different fatty acids were determined in seed oil. The oleic acid (74.5%), palmitic acid (8.3%) and stearic acid (C18:2, 5%) were the major fatty acids identified. High oleic acid content also proved that *Moringa oleifera* Lam oil should be considered a fatty acids source with great potential for biofuels production.

Keywords: *Moringa oleifera* Lam; vegetable oil; fatty acids; oleic acid.

COMPOSIÇÃO CENTESIMAL E PROPRIEDADES FÍSICO-QUÍMICAS DO ÓLEO EXTRAÍDO DE SEMENTES DE *MORINGA OLEIFERA* LAM CULTIVADAS NO ESTADO DE SERGIPE, BRASIL.

Resumo: Neste estudo, foram investigadas a composição centesimal das sementes e as propriedades físico-químicas do óleo extraído das sementes de *Moringa oleifera* Lam. Os resultados da composição centesimal mostraram que a semente consistia em 41% de óleo bruto, 32,4% de proteínas, 4,2% de cinzas, 19,54% de carboidratos e 2,86% de umidade. Esses dados indicam que as sementes de *Moringa oleifera* Lam, ricas em nutrientes, são um potencial ingrediente para produtos alimentares dietéticos e saudáveis. Além disso, nove ácidos graxos diferentes foram determinados no óleo. O ácido oleico (C18: 1, 74,5%), o ácido palmítico (C16: 0, 8,3%) e o ácido esteárico (C18: 2, 5%) foram os principais ácidos graxos identificados. O alto teor de ácido oleico também comprovou que o óleo de *Moringa oleifera* Lam deve ser considerado uma fonte de ácidos graxos e tem grande potencial para a produção de biocombustíveis.

Palavras-chave: *Moringa oleifera* Lam; óleo vegetal; ácidos graxos; ácido oleico.

1. INTRODUCTION

Moringa oleifera Lam is a native species from northern India. Due to high adaptability in nutrient-poor soils, this specie were spread in several regions worldwide, such as the Brazilian northeast [1]. *Moringa oleifera* Lam seed oil is composed of fatty acids equivalent to olive oil and has been applied to biofuels manufacturing [2,3]. The industrial application of vegetable oils requires an analysis of seeds conservation status, as well as the physicochemical properties of oil extracted [4]. However, information on the centesimal composition and physicochemical properties of oil extracted from *Moringa oleifera* Lam seeds is lacking. In the present study, the centesimal composition seeds and physicochemical properties of oil extracted from *Moringa oleifera* Lam seeds were studied. The fatty acid compositions of oil were analyzed using gas chromatography (GC). In addition, the extracted oil was analyzed by Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TG/DTA).

2. METHODOLOGY

2.1. Centesimal composition

The centesimal composition of *Moringa oleifera* Lam seeds were determined by humidity, ash, lipids, proteins and carbohydrates contents, according to the Official Methods of Analysis [5].

2.2. Oil extraction

The oil from 50 g of ground *Moringa oleifera* Lam seeds was extracted with n-hexane using a Soxhlet apparatus for 8 h at 68 °C. Then, solvent was removed from oil using a rotary evaporator (IKA, RV 10 Control). Finally, the oil was oven-dried at 60 °C to completely remove all residual solvent traces. The oil was stored under refrigeration at approximately 4 °C.

2.3. Oil characterizations

The physicochemical properties: acid value, free fatty acid content, iodine value, saponification value, peroxide value, density kinematic viscosity and refractive index, were determined according to standard analytical methods recommended by Official Methods of Analysis [5].

The fatty acid compositions of oil were analysed by gas chromatography/quadrupole mass spectrometry (GC/qMS). The samples were analysed on a GC/qMS instrument (QP2010 plus, Shimadzu, Tokyo, Japan) equipped with an AOC20i auto-injector (split/splitless mode). The chromatographic separation was performed using a DB-5 column (methyl silicone with 5% phenyl groups – 60 m × 0.25 mm id × 0.25 µm film thickness). The oven temperature was programmed as follows: the column was held at 50 °C for 2 min; heated to 200°C at a rate of 5 °C min^{-1} and then to 230 °C at 10 °C min^{-1}; held at 230 °C for 5 min; heated to 245 °C at a rate

of 2° C min^{-1} and then to 300 °C at 10 °C min^{-1}; and finally held at this final temperature for 10 min. All data were analysed by the GCMS solution software 2.6 (Shimadzu, Japan), and the compounds were tentatively identified using the NIST-05 spectral library database.

Extracted oil was additionally analyzed by Fourier transform infrared spectroscopy (FTIR) analysis (BOMEMMB-100 FTIR spectrophotometer), with a resolution of 4 cm^{-1}, from 500 to 4000 cm^{-1}. Thermogravimetric analysis (Shimadzu DTG-60H simultaneous DTA-TG apparatus) were carried out in synthetic air, *Moringa oleifera* Lam oil (5 mg) was heated from 25 to 600 °C at a rate of 10 °C min^{-1} with a gas flow rate of 50 mL min^{-1}.

3. RESULTS AND DISCUSSION

3.1. Centesimal composition

The centesimal composition of the *Moringa oleifera* Lam seeds after grinding and sieving is shown in Table 1. Nutritional composition of *Moringa oleifera* Lam seeds (in dry basis), was 2.86% ± 0.3 for humidity, 14.2% ± 0.2 for ash, 41% ± 0.2 for lipids, 32.4% ± 0.8 for proteins and 19.54% ± 1.3 for carbohydrates, which are in agreement with previous studies reported [6,7]. Small variability may be due different temperature and availability of water during seeds maturation stage.

Table 1. Centesimal composition of *Moringa oleifera* Lam seeds.

Centesimal composition	Determined values	Abiodun et al. (2012) [6]	Bridgemohan et al. (2014) [7]
Humidity (%)	2.86 ± 0.3	4.7	5.4
Ash (%)	4.2 ± 0.2	4.1	3.7
Lipids (%)	41 ± 0.2	45.8	38.2
Proteins (%)	32.4 ± 0.8	28.0	37.2
Carbohydrates (%)	19.54 ± 1.3	18.3	15.5

Values are mean ± SD of triplicate determinations

3.2. Oil characterization

Table 2 summarizes the physicochemical properties of *Moringa oleifera* Lam oil extracted and reported by different authors. The acid value of *Moringa oleifera* Lam oil was 21.41 mgKOH/g, similar to acid values reported in literature [3,8]. The high acidity index obtained indicates that oil has undergone hydrolysis of triacylglycerol chains, releasing free fatty acids. The determination of iodine index is a useful measure for understanding the degree of oil unsaturation and is directly related to their oxidative stability [9]. The average value obtained (72.64 gI_2/100g) is not significantly different

from values reported by other authors [3,8]. Saponification index was 181.99 mg_{KOH}/g, within the expected range for vegetable oil seeds or saponifiable animals (180 and 200 mg_{KOH}/g) [8]. The peroxide value was 3.98 $meqO_2/kg$, which indicates high resistance to oxidation of *Moringa oleifera* Lam oil [10].

In evaluation of physical properties, refraction index was 1.45, whereas density at 15 °C amounted to 894.5 kg/m^3 and kinematic viscosity at 40 °C was 40.32 cSt. These values are consistent with the requirements of standard for vegetable oils [11]. The content of water in oil determined using Karl Fischer method amounted to 0.35%. This low moisture content may be related to seed storage or interaction with the solvent during the oil extraction process. The variations observed in Table 2 can be attributed to the different environmental conditions, processing and handling seeds.

Table 2. Physicochemical properties of oil extracted from *Moringa oleifera* Lam seeds.

Properties	Determined values	Bhutada et al., (2016) [3]	Pereira et al., (2016) [8]
Acid value (mg_{KOH}/g)	21.41 ± 0.86	26.22	20.50
Free fatty acid (mg_{KOH}/g)	10.70 ± 0.86	13.11	10.25
Iodine value ($gI_2/100g$)	72.64 ± 1.24	75.06	70.70
Saponification value (mg_{KOH}/g)	181.99 ± 2.62	172.16	179.40
Peroxide value ($meqO_2/kg$)	3.98 ± 0.26	ND	5.40
Density at 15 °C (kg/m^3)	894.5 ± 5.51	240	907.20
Kinematic viscosity at 40 °C (cSt)	40.32 ± 0.28	ND	39.10
Refractive index	1.45 ± 0.04	ND	ND
Water content	0.35 ± 0.02	ND	ND

Values are mean ± SD of triplicate determinations

ND = not determined

Fatty acid composition of obtained *Moringa oleifera* Lam oil is presented in Figure 1(A). *Moringa oleifera* Lam oil consists mainly of oleic acid (C18:1, 74.5%), followed by palmitic acid (C16:0, 8.3%) and stearic acid (C18:2, 5%). The content of myristic acid (C14:0) equal to 1%, linoleic acid (C18:2) 2.5%, arachidic acid (C20:0) 3%, gadoleic acid (C20:1) 0.7, behenic acid (C22:0) 2% and lignoceric acid (C24:0) 3%. The total of saturated and unsaturated fatty acid was 22.3% and 77.7% respectively. The fatty acid profile demonstrates that *Moringa oleifera* Lam oil can be an excellent source of oleic acid which gives a high oxidative stability to oil for several applications [3,10]. In addition, oleic acid is one of the most effective lubricating additives [12,13].

ISSN: 2357-7592

Figure 1. Analysis of *Moringa oleifera* Lam oil: (A) Fatty acid composition (B) FTIR spectra (C) TG/DTA.

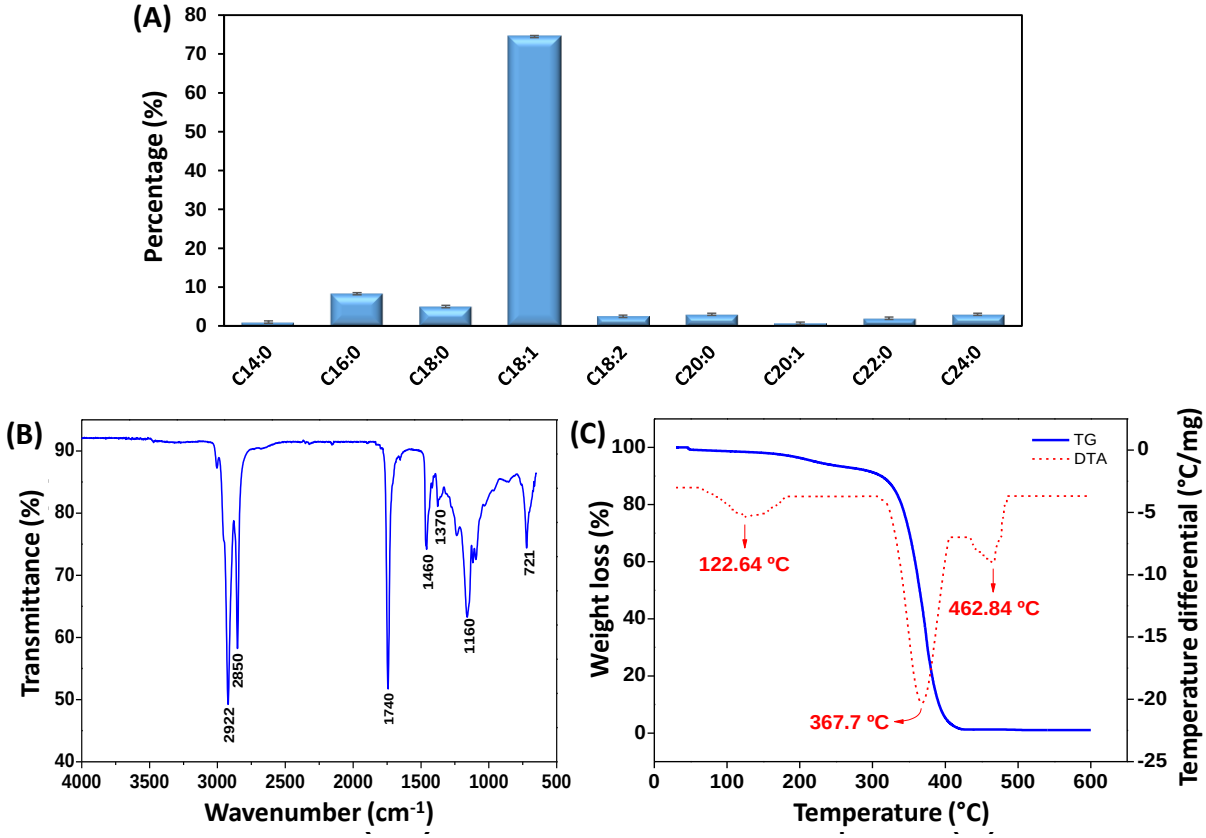

Figure 1(B) shows FTIR spectrum used to analyze the main functional groups present in the oil extracted from *Moringa oleifera* Lam seeds. The absence of a peak after 3000 cm^{-1} indicates very low concentrations of impurities contained in hydroxyl groups (OH), such as free glycerol and water [14], which corroborates the moisture data (0.35%) obtained in this study. There is a wide band from 2922 to 2850 cm^{-1}, due to the symmetrical and asymmetric elongation of the C–H group, attributed to the fatty acids present in the oil [15]. In the region between 1770 and 660 cm^{-1}, there are intense bands attributed to the elongation of the C=O bond, a characteristic carbonyl group of proteins and fatty acid structures. The peak in the region 1460-1370 cm^{-1} indicates the presence of C–C. The peaks at 1160 and 721 cm^{-1} are assigned to C–O bonds and the asymmetric deformation of CH_2 group present in fatty acids, respectively [15].

The thermogravimetric curve and its derivative (TG/DTA) show the volatilization and/or thermal combustion of 100% of the mass of the oil in three thermal stages (Figure 1(C)). The first stage, starting at approximately 30 °C and finishing at 181 °C, where a loss of mass of 0.38% associated with the volatilization of the water contained in the oil. The second stage occurred between temperatures 181 and 405 °C with weight loss equal to 93.03%. In this stage, the greater decomposition indicates the decomposition of fatty acids in the oil, for example oleic acid, which has a boiling point of 360 °C. The third stage is associated with the decomposition of the existing impurities in the oil constituents, showed a weight loss of 6.54% at temperatures between 405 and 500 °C [16].

4. CONCLUSION

In this study, the chemical compositions of oils from *Moringa oleifera* Lam seeds were identified, and the main components were proteins, lipids and carbohydrates. The physicochemical properties of oil from *Moringa oleifera* Lam seeds were also determined following extraction either with *n*-hexane. Oleic acid is the major constituent of *Moringa oleifera* Lam oil (74.5%), been a promising source of vegetable oil for several industrial applications.

5. REFERENCES

[1] LEONE A, SPADA, A, BATTEZZATI A, SCHIRALDI A, ARISTIL J, BERTOLI S. Cultivation, genetic, ethnopharmacology, phytochemistry and pharmacology of *Moringa oleifera* leaves: An overview. **International Journal of Molecular Sciences**, v. 16, p. 12791–835, 2015.

[2] DE SOUZA MC, DE SOUZA GONÇALVES JF, GONÇALVES PC, LUTIF SYS, DE OLIVEIRA GOMES, J. Use of Jatropha and Moringa oils for lubricants: metalworking fluids more environmental-friendly. **Industrial Crops and Products**, v. 129, p. 594-603, 2019.

[3] BHUTADA PR, JADHAV AJ, PINJARI D V., NEMADE PR, JAIN RD. Solvent assisted extraction of oil from *Moringa oleifera* Lam seeds. **Industrial Crops and Products**, v. 82, p. 74–80, 2016.

[4] BELGACEM MN, GANDINI A. Materials from vegetable oils: major sources, properties and applications. **Monomers, Polymers and Composites from Renewable Resources**, v. 32, p. 39–66, 2008.

[5] ASSOCIATION OF THE OFFICIAL ANALYTICAL CHEMISTS (AOAC). Official Methods of Analysis of AOAC International - 20th Edition, 2016. 20th ed. Gaithersburg: AOAC. 2016.

[6] ABIODUN O, ADEGBITE J, OMOLOLA O. Chemical and physicochemical properties of Moringa flours and oil. **Global Journal of Science Frontier Research**, 2012.

[7] BRIDGEMOHAN P, BRIDGEMOHAN R, MOHAMED M. Chemical composition of a high protein animal supplement from *Moringa oleifera*. **African Journal of Food Science and Technology**, 2014.

[8] PEREIRA F, SCHULER A, SILVA A, GALVÃO C, PEREIRA R, FILHO N. Turbidity and acidity as monitoring parameters in the purification of *Moringa oleifera* oil for biodiesel production. **American Chemical Science Journal**, v. 16, p. 1–9, 2016.

[9] SÁNCHEZ-MACHADO DI, LÓPEZ-CERVANTES J, LÓPEZ-HERNÁNDEZ J, PASEIRO-LOSADA P. Effect of the refining process on *Moringa oleifera* seed oil quality. **Food Chemistry**, v. 187, p. 53–7, 2015.

[10] RAHMAN IM, BARUA S, RAHMAN MA, HASEGAWA H. Physicochemical properties of *Moringa oleifera* Lam seed oil of the indigenous-cultivar of Bangladesh. **Journal of Food Lipids**, v. 16, p. 540–53. 2009.

[11] Codex Alimentarius. Standard for Named Vegetable Oils. CODEX STAN 210-1999 (Ammended 2015). 2015.

ISSN: 2357-7592

[12] JI M, LIU S, XIAO H. Tribological behaviors of water-based drilling mud with oleic acid-filled microcapsules as lubricant additives for steel-steel contact. **Industrial Lubrication and Tribology**, 2020.

[13] DOIG M, WARRENS CP, CAMP PJ. Structure and friction of stearic acid and oleic acid films adsorbed on iron oxide surfaces in squalane. **Langmuir**, v. 22, p. 34–42, 2014.

[14] IVANOIU A, SCHMIDT A, PETER F, RUSNAC LM, UNGUREAN M. Comparative study on biodiesel synthesis from different vegetables oils. **Chemical Bulletin**, v. 56, p. 94–8, 2011.

[15] ARAÚJO CST, ALVES VN, REZENDE HC, ALMEIDA ILS, DE ASSUNÇÃO RMN, TARLEY CRT. Characterization and use of *Moringa oleifera* seeds as biosorbent for removing metal ions from aqueous effluents. **Water Science and Technology**, v. 62, p. 2198–203, 2010.

[16] RASHID U, ANWAR F, ASHRAF M, SALEEM M, YUSUP S. Application of response surface methodology for optimizing transesterification of *Moringa oleifera* oil: Biodiesel production. **Energy Conversion and Management**, v. 52, p. 3034–42, 2011.

ISSN: 2357-7592